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Development of a Quadrupole-Based Secondary-Ion Mass Spectrometry (SIMS) System at Lewis Research Center

Carlos Vargas-Aburto
Kent State University
Kent, Ohio

Paul R. Aron
Lewis Research Center
Cleveland, Ohio

and

Dale R. Liff
Kent State University
Kent, Ohio

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DEVELOPMENT OF A QUADRUPOLE-BASED SECONDARY-ION

MASS SPECTROMETRY (SIMS) SYSTEM

AT LEWIS RESEARCH CENTER

Carlos Vargas-Aburto
Kent State University
Kent, Ohio 44242

Paul R. Aron
National Aeronautics and Space Administration
Lewis Research Center
Cleveland Ohio 44135

and

Dale Liff
Kent State University
Kent, Ohio 44242

SUMMARY

We report the design, construction, and initial use of an ion microprobe to carry out secondary ion mass spectrometry (SIMS) of solid samples. The system is composed of a differentially pumped custom-made UHV chamber, a quadrupole mass spectrometer and a telefocus A-DIDA ion gun with the capability of producing beams of Cesium, as well as of inert and reactive gases. The computer control and acquisition of the data have been designed and implemented using a personal computer with plug-in boards, and external circuitry built as required to suit the system needs. The software is being developed by us using a FORTH-like language. Initial tests aimed at characterizing the system, as well as preliminary surface and depth-profiling studies are presently underway.

INTRODUCTION

Secondary ion mass spectrometry (SIMS) is an analytical technique in which charged particles (secondary ions) emitted from a surface subjected to bombardment by energetic (primary) ions are mass analyzed and detected (ref. 1). Typically, positive ions are accelerated and focused onto a solid target. A fraction of (sputtered) secondary ions pass through an aperture into a mass spectrometer tuned to a specific mass-to-charge ratio (m/e), where they are analyzed. Individual pulses from the detector are counted for a certain time, and then the next m/e ratio is selected. Hence, typical SIMS raw data consists of plots of intensity (counts per unit time) versus mass per unit charge.

In recent years, SIMS has established itself as one of the most important techniques used to do materials characterization (ref. 2). Auger electron spectroscopy (AES), x-ray photoelectron spectroscopy (XPS), ion scattering spectroscopy (ISS), and Rutherford backscattering spectrometry (RBS), are other techniques typically used for surface analysis (ref. 3). Each of these techniques has its advantages and disadvantages, and careful consideration of the

nature of a particular problem is required to select the technique(s) best suited for it. In what follows, the discussion will focus on SIMS and its implementation in this laboratory.

In SIMS, a small fraction of the products emitted from the sample as a result of the primary ion bombardment are in the form of both positively and negatively charged ions. Their relative abundance is the result of the physical and chemical properties of the interacting systems (primary ion and sample). Therefore, depending on the sample under study and the experimental conditions, it may be desirable to monitor either positive or negative charged particles. The first case is commonly known as positive SIMS and the second as negative SIMS. In each case, two modes of operation are usually recognized: static SIMS and dynamic SIMS (ref. 4). The difference between the two modes results from the amount of damage caused to the surface under study. Briefly, static SIMS refers to analyses where the primary current density is low enough (typically, 1 nA/cm² or less) that, on the average, no more than one primary ion hits any given location on the sample surface during the analysis time. When doing dynamic SIMS, erosion of the sample surface can take place at rates as high as a few micrometers per hour, depending on the ion source intensity and focusing capabilities. Measurement of the concentration as a function of sample depth (in-depth profiles) for selected (atomic and/or molecular) fractions can then be obtained. The technique can detect all elements, including hydrogen, and their isotopes. SIMS has a particularly high sensitivity. Under suitable conditions and depending on the sample under study, detection limits in the ppb range are often obtained; this fact makes it unique among the surface analysis techniques and particularly attractive for the study of dopants and impurities found in materials (refs. 5 and 6).

We describe here the design and construction of a SIMS system (ref. 7) built primarily to study and characterize samples of interest to photovoltaic researchers. The construction of the system started in the summer of 1986 and after roughly 3 years it is fully operational. A significant amount of time has been spent in implementing and properly coordinating the performance of the various components of the system. Careful consideration has been given to virtually every constituent, from the vacuum components to the electronic instrumentation needed for analysis. Further additions and improvements to the present configuration can be made, and some are in the process of being implemented, as discussed in the section on conclusions.

SYSTEM OVERVIEW

A photograph of the SIMS system is shown in figure 1. A simplified diagram showing the major components is presented in figure 2. The custom-built analytical (main) chamber is made of stainless steel and is shaped as a cylinder, 8 in. in (internal) diameter and 12 in. high. A total of 16 ports, at two different levels, are available on the body of the chamber. Approximate volume of the chamber including the ports is roughly 20 liters.

In the upper level, four 6 in. ports are located at 90° intervals (see fig. 3). One of them (P1) is used as a viewport; the other three are used for the transfer chamber (P4), the mass spectrometer (P3) and the ion gun (P4)). As shown in figure 3, the other 12 are 2-3/4 in. ports which are located symmetrically around the body of the chamber. One of them (P5) is tilted and

points to the intersection (T) of the P1-P3 and P2-P4 axes. The top flange provides a 6 in. port for the sample manipulator and a tilted 2-3/4 in. port (P6) that also points to T. The bottom of the chamber provides access (P7) to the main vacuum pump, through a manually-operated UHV gate valve.

The chamber is evacuated by an air-cooled 8 in. cryogenic vacuum pump (CRYO-TORR 8, CTI Cryogenics) with a pumping speed of 1500 liter/sec for air. Total volume pumped by the main cryo pump adds to about 30 liters, which includes the sample chamber, the volume occupied by the quadrupole assembly and other dead spaces. Manual gate valves allow the chamber to be isolated from the cryo pump, the transfer chamber and the ion gun. Only three 2-3/4 in. ports are presently under use. One contains feedthroughs to an electron flood gun filament; another is used for bakeout of the chamber via an internal filament, and the third port has an up-to-air bellows valve (4BW, Nupro), normally attached to a gaseous nitrogen source.

The transfer chamber is pumped by two sorption pumps (2361510, Perkin-Elmer) which were chosen to minimize contamination and vibration. The chamber is equipped with a 6 in.-hinged viewport flange and allows the mounting of the sample holder at the end of a magnetic linear drive which is used for sample transfer to the main chamber (fig. 2). A gate valve isolates the transfer chamber from the roughing pumps, and both a thermocouple gauge and an ion gauge serve to monitor the pressure.

The ion gun is differentially pumped through a 2 mm orifice by an air-cooled turbomolecular pump (TPU-170, Balzers) with a pumping speed of 170 liters/sec for nitrogen. A rotary vane mechanical pump (D4A, Leybold-Heraeus) backs the turbo pump. An adsorption trap is located between the turbo and the mechanical pump to reduce backstreaming.

The operation of the ion gun requires that the source be floated to an operating voltage anywhere from 0.1 to 15 kV while the accelerating electrode is maintained at ground potential. As a result, an insulating box (plexiglas) completely surrounds all the high-voltage portions of the source and of the gas handling system (see below). A safety interlock automatically cuts power to the high voltage supply should the door to the box be opened.

The gas-handling system is also shown in figure 2. A manifold is used to switch among the various gases used in the ion gun. Three gas sources are connected to the system; ultra-high purity nitrogen, argon and oxygen are normally used, but any other gas source can be readily connected. With proper manipulation of the valves, any of the three gases can be fed to the ion source. In order to ensure a high pressure stability in the latter, a 2-step pressure drop scheme is utilized. A first two-stage regulator maintains a constant pressure of about 1 bar. A second regulator further reduces this to 0.5 bar in the reservoir to which the input port of a variable leak valve (203, Granville-Phillips) is connected. In operation, gas is admitted into the ion source at a constant rate, via this valve. Because of its location, the valve floats at the acceleration potential. Hence, an insulating arm allows for control of the valve flow rate. The gas-handling system is maintained at ground potential by the use of a glass-to-metal adapter placed in-line just ahead of the leak valve.

Dry nitrogen gas is normally used for purging the system. If desired, purging can be done independently for the ion gun, the main chamber and the transfer chamber. Additionally, the system can be purged with argon gas; this is necessary when the cesium source is in operation.

The ultimate residual gas pressure within the main chamber is typically less than 5×10^{-10} mbar with no prior bakeout. With no gas being admitted to the ion source, the base pressure in the latter is typically an order of magnitude higher than in the main chamber as measured by an ion gauge located at the high vacuum side of the turbo pump. During normal operation, the ion source pressure is kept at $1-2 \times 10^{-6}$ mbar.

The positioning of the sample within the main chamber is accomplished using a UHV-compatible XYZ-and-rotary precision manipulator (PM-600, Huntington) with a linear resolution of 2 μ m in any direction, and a 360° rotation capability and angular resolution of 0.1°. Maximum linear coaxial travel in the X or Y direction is ± 12.5 mm.

Ion Gun

The ion gun used in the system (Atomika Inc., WF-610) is the type used in the ion microprobe developed by Wittmack (A-DIDA 3000). A detailed review of its operation and performance can be found in a number of publications by Wittmack and others (ref. 1 and references therein). Specifications for the gun, as published by the manufacturer, are summarized in appendix A.

The ion gun is composed of four major elements. The plasma ion source, the beam extraction and the accelerating electrodes, the mass (Wien) filter, and the beam-forming system. A schematic of the gun is presented in figure 4. Only the major elements have been identified and labeled. Approximate limits on the distance "a" from the ion gun to the electro-optical assembly, and "f", from the latter to the sample, are 100 to 500 mm and 30 to 100 mm, respectively.

A more detailed view of the gun and its internal structure is shown in figure 5. The first set of X and Y electrodes are primarily used to steer the beam out of the acceleration-section of the vacuum system; they are controlled by 10-turn potentiometers on the front panel of the Beam Align module (see below). The Y plates play a double role, as they also serve to set the electrostatic cross field needed by the Wien filter to mass-select the primary ions. The N electrodes deflect the ions through an angle of about 2°; this causes the neutral fraction to miss the entrance aperture A6. Finally, the second set of XY electrodes are used to raster scan the beam which then enters a focusing electrostatic (Einzel) lens.

The plasma ion source permits the use of a hot or a cold cathode. The first is used for the generation of ion beams of inert gases. The latter is normally used for oxygen and other reactive gases. A cesium ion source (Atomika, Inc., 3-21) can be readily substituted by the plasma source and attached to the main body of the gun (where the extraction and accelerating electrodes are located). It consists of a frit-type thermal ionizer with a cesium reservoir and extraction and cooling system, and a rack-mounted floating power supply for frit-heating and monitoring.

The plasma ion gun requires four components for its operation. The ion gun control (IGC) unit, the beam alignment (BA) power supply, the electro-optical assembly (EA) and the scanning control (SC) unit. Of these, three are rack-mounted, except for the EA which is located inside the main chamber. For the cesium source, an additional module, the cesiums gun control (CsGC) unit is also needed.

The IGC unit supplies the voltages and currents needed for the operation of the ion gun in both the hot- and cold-cathode modes. Four parameters can be adjusted, namely, the filament current (used only in the hot-cathode mode), the discharge voltage and current which set the conditions for the plasma discharge, the magnet current, which supplies the current for the electromagnet, and the extraction voltage, which controls the extraction field for generation of the ion beam.

Figure 6 shows the A-DIDA gun configured for hot cathode operation. Connections are shown for the electron-emitting filament, the magnetic-field producing coil, the discharge current and the extraction electrode. The extraction and the accelerating electrodes act as an immersion lens. The ions are given a fraction (1/30) of their final energy by the extraction electrode; the acceleration electrode then supplies the additional amount.

As stated above, the BA module controls the deflection fields applied to the ion beam via the XY electrodes, to steer it past aperture A4 (see fig. 5). Furthermore, it is through these electrodes (Y) that the primary ion beam can be blanked, i.e., prevented from reaching the sample, by applying a 300 V reverse bias to them. Under these conditions, the beam will not go through aperture A4. As discussed later in detail, blanking of the ion beam must be performed at specific times when sputtering a sample's surface. Finally, the BA module also supplies the field that steers the ion beam into apertures A5 and A6, thereby stripping it from background ions and neutrals.

The EA is the last stage in the path of the primary ion beam towards the sample. It is in the main chamber, and consists of a beam-limiting aperture, beam-deflection electrodes, the Einzel lens (ref. 8), and an exit aperture. A voltage of the order of the acceleration voltage is applied to the Einzel lens and adjusted to obtain the degree of focusing desired.

The SC unit provides the deflection voltages needed to raster scan the ion beam on the sample surface. The raster signal controls the position of the beam on the sample surface by moving it in a step-wise fashion on a square pattern of 200 by 200 points (one frame). The final amplitude of the scan on the sample, which depends on geometry and other parameters, can be varied from zero up to a few square millimeters via a 10-turn potentiometer.

The center point of the area of the sample to be scanned can be adjusted in the X and Y directions by two 10-turn potentiometers. A frame can be scanned at rates going from 1 down to 0.01 frame/sec, in increments of 0.01 frame/sec. The unit also allows for selection of continuous- or single-scan modes via a toggle switch. Reset and start pushbuttons are also available for control of the beam when in the single-scan mode of operation. The controls for all these functions are found in the front panel of the SC unit.

The SC unit also produces an electronic signal, in synchronism with the raster pattern, that is normally used to suppress the secondary ion signal coming from the edges of the rastered area.

An interface is available at the rear of the SC unit to permit computer control of its operation. In a later section a description is presented of the implementation of this mode of operation via a personal computer and additional hardware and software.

Secondary Ion Detection

Figure 7 shows a schematic of the secondary-ion detection subsystem. The first element is the axial energy analyzer (Extrel, 616-1) or "Bessel Box," which selects (transmits) those secondary ions within a window in their energy distribution and directs them to the entrance aperture of the quadrupole assembly with the help of two additional focussing electrodes. The controls and associated electronics (Extrel, 275-E5) are also shown in the figure, including those for an electron flood gun. The various voltages, as they are used to control the transmission and focussing of ions to be mass analyzed, are clearly identified.

The mass spectrometer consists of a quadrupole assembly with a 1.6 cm (5/8 in.) pole diameter (Extrel, 7-270-9). When used in conjunction with its quadrupole power supply (Extrel, 011-1) and the High-Q heads presently available in our laboratory (Extrel, 14 at 3.9 MHz, and 11(B) at 1.5 MHz), a maximum m/e of 367 can be reached. The ion signal is detected by an electron multiplier (Galileo, CME-4816) used in the pulse counting mode.

The quadrupole power supply comprises two modules: the quadrupole control (QC) and the rf power supply. The QC gives access to all the parameters needed during normal operation of the mass spectrometer, e.g., resolution, pole bias, mass range, rf/dc balance, etc, and it allows monitoring of one mass range which can be set by dials (initial mass and range of masses to be swept) in the front panel. An additional module, a mass programmer (Extrel, 091-3), allows up to six mass ranges to be monitored automatically in a sequential fashion, and also lets the user select the time per sweep (0.003 to 1000 sec in discrete increments) and the mode of operation: single or continuous scan. This module is normally used during mass calibration procedures.

The detection of positive or negative secondary ions requires the reversal in sign of the dc voltages associated with the energy filter and focusing electrodes prior to entering the quadrupole assembly. This is accomplished via a front-panel switch on the 275-E5 unit. However, a reverse biasing is also required for the electron multiplier. In the positive mode, the collector of the multiplier is biased (negative) to attract the positive ions. In the negative mode, the entrance to the detector must be biased positive. In both cases, the secondary electrons must see an increasingly positive potential in their path towards the anode of the multiplier.

For positive SIMS operation, a pre-amplifier/electrometer module (Extrel, 031-2) would be used to provide both the bias voltage for the electron multiplier and power for the operation of the preamplifier. In the present configuration, which allows for both positive and negative SIMS operation, the

preamplifier/electrometer module is used only to monitor the beam current, and two high voltage power supplies (Bertan, 355 and 320P) provide the required biasing for the electron multiplier.

Figures 8 and 9 show the biasing scheme for the positive and negative modes of operation, respectively. Suitable high voltage power supplies are used to provide HV⁺ and HV⁻, depending on the operating mode. The back flange of the mass spectrometer, with all the connections needed for the detector, the quadrupole assembly, and the energy filter and ionizer, is shown in figure 10. Figure 11 shows the connections to every one of these elements, as seen from the vacuum side. For completeness, figure 12 shows the internal connections when in the "total ion" mode, i.e., detection of net ion current as measured without amplification by the electron multiplier.

Imaging has also been implemented in the SIMS system. This has been accomplished by modulating the Z-axis of an oscilloscope with the signal from the output of the ion detector. Voltages generated by the SC unit are used to drive the horizontal and vertical scales of an oscilloscope. These signals are proportional to the voltages applied to the beam scanning electrodes. Hence, a two-dimensional map of any detected (positive or negative) ion is obtained. Absorbed-current images are also obtained by amplifying the net current to the sample and applying it to the z axis.

DATA ACQUISITION AND CONTROL

In this section a description is presented of the implementation of computer-based data acquisition and control of the SIMS system. The discussion focuses on the control of both the SC unit of the ion gun and the QC unit of the mass spectrometer. Ongoing work aimed at enhancing the performance of the system via additional control and data processing and display is summarized in the last section.

Ion Beam Control

Control of the primary ion beam via the SC unit can be exerted at a number of levels. The decision was made to implement it to the level where no modification of the timing section of the SC unit would be required. Hence, the design focused on providing the start, reset and scan mode (single/continuous) signals to the unit. In this scheme it is necessary to turn the beam off during setup times by means of the BA module.

Figure 13 presents a block diagram of the SC unit. The output of a 2 MHz clock is used to produce a square wave signal that drives two 8-bit binary counters, whose output is independently converted to analog voltages. Slow (S) and fast (F) voltage ramps are thus generated which are then offset and amplified to drive the X (horizontal) and the Y (vertical) beam scanning electrodes. Comparison of these voltages with the preset value defined by the GATE thumbwheel switch is used to generate the signal to electronically gate the incoming data. As shown in figure 13(b), a line scan through the center of the frame can also be performed by supplying appropriate inputs to the decoder. Outputs to drive an oscilloscope in the XY-mode are also generated, which are available in the 50-pin connector at the rear of the unit.

When in the single-scan mode, the start signal triggers the scanning of the sample surface by the primary ion beam. A total of 200 lines are digitally scanned, 200 points to a line, at the end of which a reset signal has to be provided for the beam to return to its original position. Figure 14(a) shows the corresponding timing diagram.

The scan pattern as well as the correlation between the beam position and the GATE signal is shown in figure 14(b). The latter is generated by the SC unit in response to the setting of the thumbwheel switch labeled GATE ($\times 10$ percent). The square of the setting on the switch represents the fraction of the time (as defined by the TIME setting) that the signal is active, i.e., that the ion beam is inside the center (gated) portion of the scanned area.

As described below, the GATE signal is used to reject the data coming from within a certain distance of the borderline of the area being scanned. This feature is of particular relevance during dynamic SIMS studies.

The digital I/O port from a high-resolution plug in card (Data Translation, DT2823) is used to generate those signals needed for control. The architecture of the board is shown in figure 15. Only three of the 16 available digital I/O lines are required to implement the desired control of the beam at this stage: start and reset, in the SC unit, and beam on/off, in the BA module. The design of the interface allows the selection of manual or computer operation of the beam. The schematic representation of the interface is shown in figure 16.

Mass Spectrometer Control

Control of the transmitted m/e by the mass spectrometer is achieved by providing a known current (0 - 1 mA) to a summing point in the QC unit.

The implementation of the interface to the computer is shown in figure 17. One of the 16-bit deglitched D/A output channels of the DT2823 board was used to provide a voltage output between 0 and 5 V. Using a precision, 10 K Ω resistor, a current within the required range is supplied to the summing point.

Pulse Counting

As described in the Introduction, in SIMS the quantity of interest is the number of secondary ions detected per unit time for given conditions of the primary ion beam and mass spectrometer. In the system described here, the signal is in the form of pulses generated by the electron multiplier.

The present configuration for data gathering is shown in figure 18. Shown explicitly are the interfaces between the PC and the ion gun, the mass spectrometer and the data acquisition module. For purposes of illustration, the measurement of the ion beam current (with a Faraday Cup) is also shown in the drawing.

The pulses from the Channeltron are pre-amplified (F-100T, MIT) into TTL-compatible levels which are then fed to a counter. During operation, the counter accepts pulses only when the GATE signal is active. At the end of a

counting period, the data are transferred to the computer via the IEEE-488 bus, for storage, display and/or further processing. The synchronization of the counter with the scanning of the beam over the sample surface presently is done by software.

A schematic representation of the configuration of the PC and associated boards is shown in figure 19.

Software Development

Development of the software needed to carry out the data acquisition and control of the SIMS system is being carried out using ASYST, a set of integrated software tools for laboratory environments (ref. 9).

The ASYST software is a commercial product which runs on personal computers (PC) that are IBM XT or AT compatible. It consists of four main modules: the ASYST language (a relative of FORTH) which includes graphics and basic statistics; a data analysis module (including matrix manipulation, curve fitting, Fourier transforms, etc); a package to support facilities to allow easy access to selected commercial or custom-built A/D, D/A and digital I/O plug-in cards and external data acquisition hardware, and finally, a package that supports the IEEE-488 interface.

Work is presently ongoing to develop a comprehensive, versatile, and user-friendly package tailored to our needs. At the present time, menu-driven software has been developed that allows the acquisition of spectra, their display on the PC monitor, the generation of plots, and the storage of the information on disk. Since these tasks are all performed within the ASYST environment, the possibility of added enhancements to the package (such as real-time display of data, etc) are within easy reach. Figure 20 shows a flow chart with the presently available options. An inverted-tree configuration characterizes the structure of the software. Access to a given branch from any other branch is done via the main menu, as indicated by the doubly-pointed arrows. By default, the software scans the lowest level from left to right, as shown by the horizontal arrows; but this can be overridden by the user.

At present, three windows are available in total at any one time. Two of them, MAIN and OPTIONS, remain unchanged throughout a run and provide access to the various modes of operation of the system. The third one is used for providing information or data relevant to the measurements to be done, or for display purposes. As an example, of the operation, part (a) of figure 21 shows the response of the program to the selection of the "SIMS" option (shown in bold type). Run parameters can be selected from built-in default values (option 1), from the previous run (option 2) or from a run previously stored in disk (option 3).

Figure 21(b) shows the response to the selection of "Setup" within the "SIMS" option. The basic information regarding the next run is requested. Notice that the bottom line of the window is used to inform the user of the type or characteristics of the expected response for each of the parameters required.

PRELIMINARY RESULTS

Although the main objective of this report is to describe the system and the approach that was followed in interfacing the various components, we present in this section some initial measurements of its properties.

Figure 22 is a plot of ion beam current as a function of acceleration voltage. The squares (\square) are data from the manufacturer. The points (O) are our measurements shortly after the gun was installed in our system. Although no exact agreement is necessarily expected, optimization of the ion source operating parameters should provide better overall agreement; in particular, at high energies.

Measurements of the beam diameter were made at two different currents by the use of a knife edge. In the first case (75 nA), the knife edge was scanned with the sample manipulator (fig. 23). The diameter, as measured from 16 to 84 percent of the signal was 48 μm . In the second case (1 nA), the beam was scanned electronically and the output of the electrometer measured as a function of time (fig. 24). The diameter at this current level was 8 μm (16 percent to 84 percent). The time base was calibrated in terms of distance by imaging a grid of known mesh size (25 μm).

These measurements are consistent with the original specifications of the ion gun. At higher energies and very low beam currents, a minimum spot size of less than 10 μm can be obtained. As expected, as the energy is lowered or the current increased, an increase in the minimum spot size is observed.

A typical residual gas analysis (RGA) spectrum of the system is shown in figure 25. The plot was generated in a two-pen color plotter linked to the PC via the IEEE-488 bus (see fig. 18), by selecting the "Display data" option at the end of the run. The usual peaks are found and labeled, but are seen to have a small offset that varies throughout the mass range. This problem, which is mostly due to inherent nonlinearities in the electronics of the mass spectrometer, is presently being addressed.

An as-obtained positive SIMS spectrum, covering an m/e range from 50 to 80, of a powder sample of iron oxide (Fe_2O_3) is shown in figure 26. The sample was pressed onto an indium foil. A 200 nm argon beam at 5 keV was used. A 1 amu interval was subdivided into 10 points (at 10 sec acquisition time per point). A 1 mm^2 frame was scanned, with data accepted from the center 35 percent of the total scanned area. The spectrum was obtained at a working pressure of 2×10^{-9} mbar.

The most prominent peaks in figure 26 are located at an m/e of 54, 56, 57 and 58. Comparison of their relative intensities (6.9, 91.7, 2.2, 0.3) shows good agreement with the natural abundances of the isotopes of iron (5.8, 91.7, 2.2, 0.3). An unidentified contribution is apparent at an m/e of 54.

A negative SIMS spectrum of a stainless steel sample is shown in figure 27. The spectrum covers an m/e range from 0 to 50. A 100-nm 5 keV argon beam was used. As is typical of most negative SIMS spectra, the background noise is higher than in positive SIMS. Comparison with similar spectra (ref. 4), shows good qualitative agreement. No attempt was made to resolve or account for possible peak interferences.

Imaging has been implemented in this system. A set of secondary-ion copper-65 images of a 3 mm 80 μm copper grid is shown in figure 28. The effect of varying the frame size is seen as it is reduced to cover roughly one mesh hole. A 15 keV 500-nA beam of singly-charged positive molecular oxygen was used as primary beam.

An example of the depth profiling capabilities of the system is shown in figure 29. An as-obtained depth profile of a 1000 Å tantalum oxide film on a tantalum substrate is presented. As shown, three different ion fragments were monitored throughout the sputtering process, and are displayed in a linear axonometric plot. The apparently broad oxide-metal interface is believed to be partly due to unwanted beam effects (variations in beam current and focussing conditions during a frame scan).

CONCLUSIONS

A SIMS system has been designed, built, and is operational. Improvements of the system are continuing, as well as efforts aimed at characterizing the system both in terms of the instrumental parameters and of sensitivities for dopants and/or impurities found in III-V semiconductors; in particular, GaAs and InP, materials that are of particular relevance to space photovoltaics.

APPENDIX A

Operating parameters for the ion gun, according to manufacturer specifications.

Ion beam energy:

0.1 to 15.0 keV

Ion beam current:

1×10^{-11} to 2×10^{-6} A

Ion Beam Current Density:

1×10^{-12} to 1×10^{-2} A/cm²

Ion beam spot size:

5 to 2000 Å

Ion beam current stability:

1 percent (1 hr), 2 percent (8 hr)

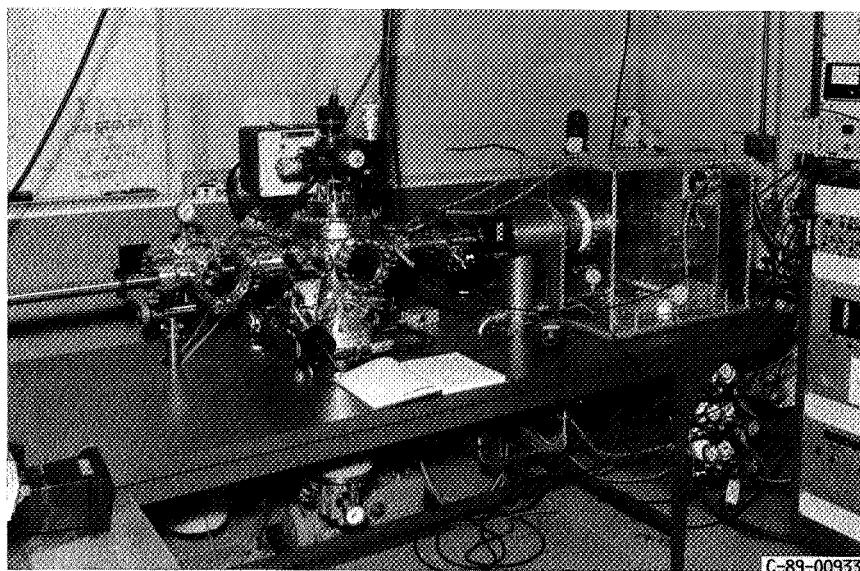
Wien filter mass resolution:

10 at 10 keV

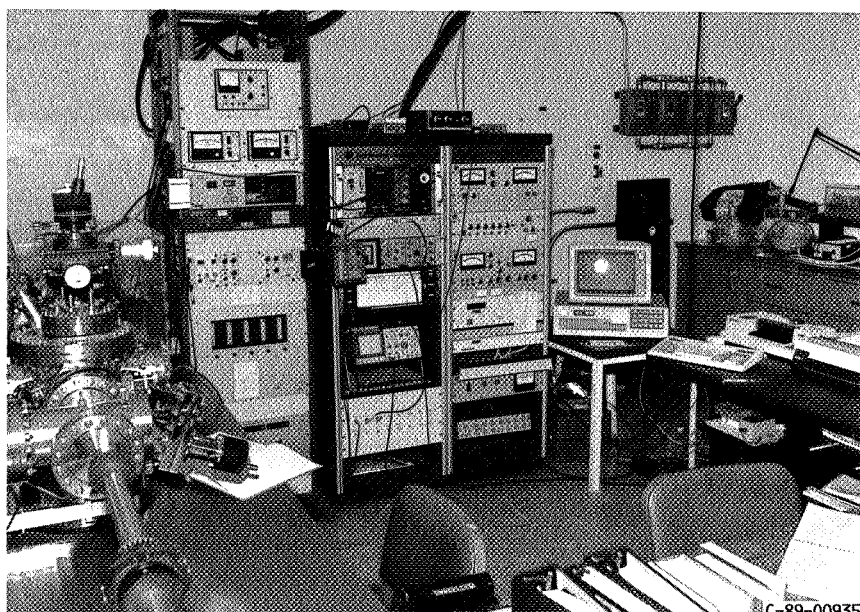
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ORIGINAL PAGE
BLACK AND WHITE PHOTOGRAPH



(a) VACUUM SYSTEM.



(b) ELECTRONICS.

FIGURE 1. - THE SIMS LABORATORY AT THE PHOTOVOLTAIC BRANCH, POWER TECHNOLOGY DIVISION, LERC (DEC. 1989).

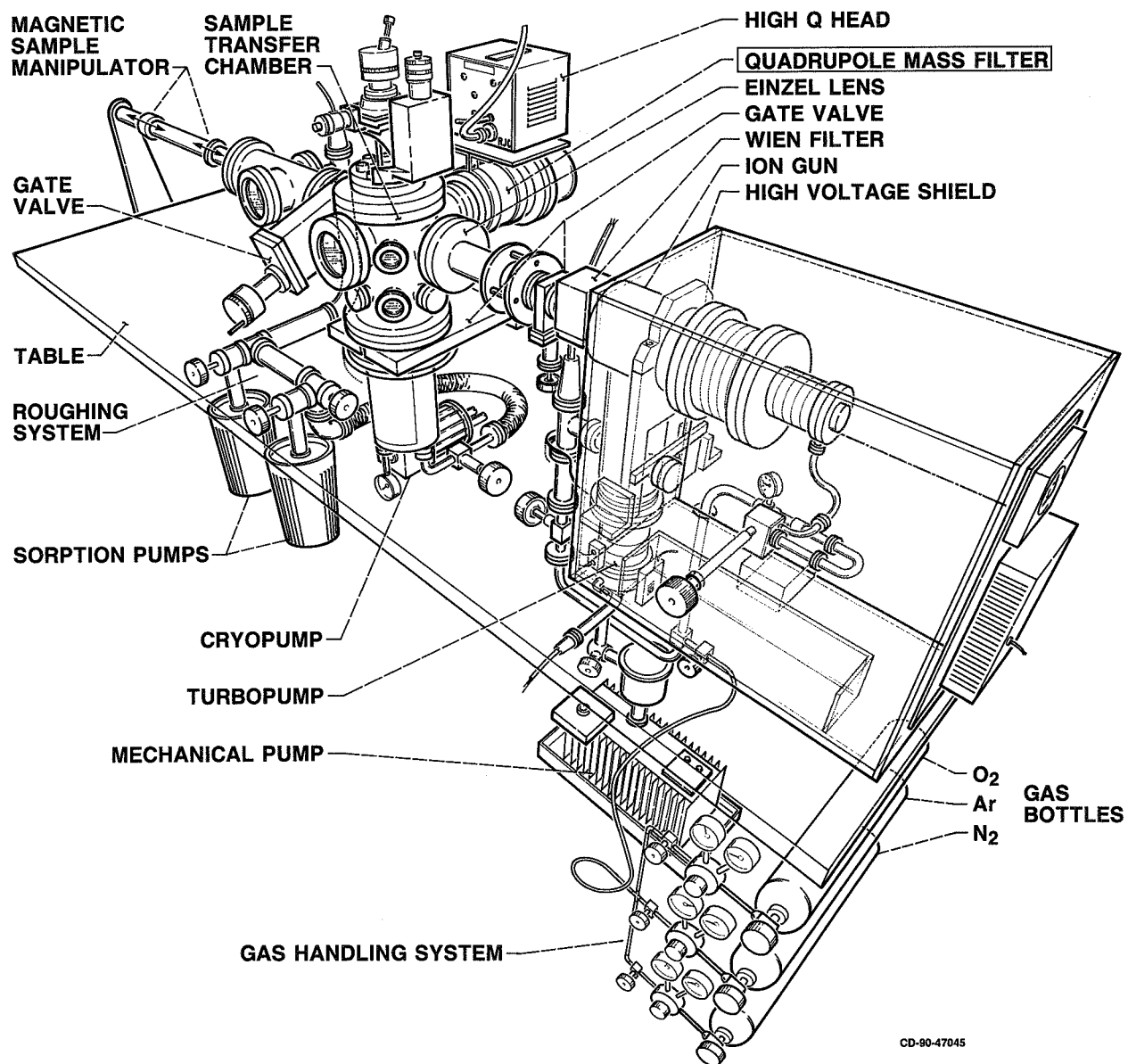
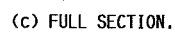


FIGURE 2. - LINE DRAWING OF THE VACUUM SYSTEM SHOWING THE MAJOR COMPONENTS.



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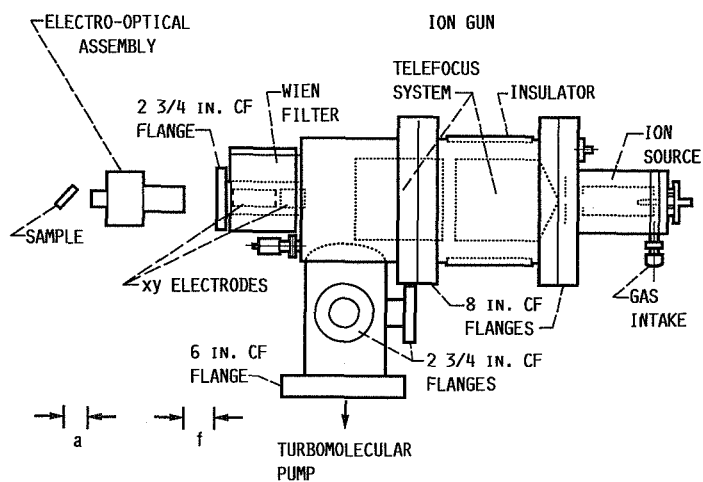


FIGURE 4. - SCHEMATIC REPRESENTATION OF THE A-DIDA ION GUN, ONLY THE MAJOR INTERNAL COMPONENTS ARE SHOWN.

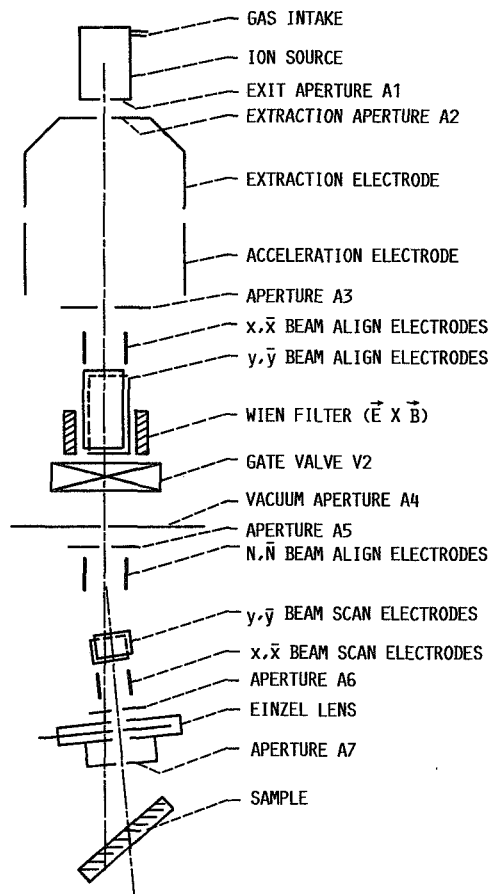


FIGURE 5. - INTERNAL ARCHITECTURE OF TELEFOCUS ION GUN A-DIDA (MODEL WF610).

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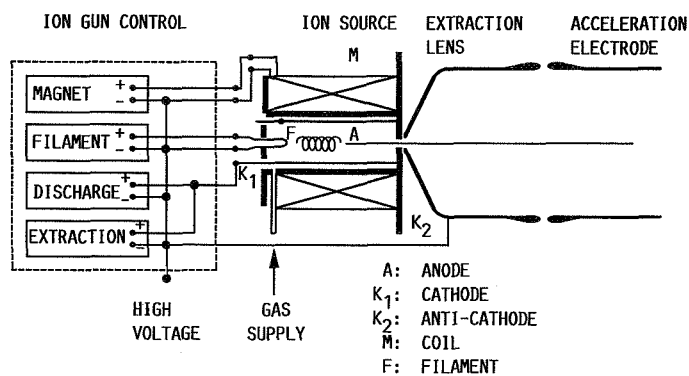


FIGURE 6. - CONTROL OF THE A-AIDA ION BEAM SOURCE WHEN OPERATED IN HOT-CATHODE MODE.

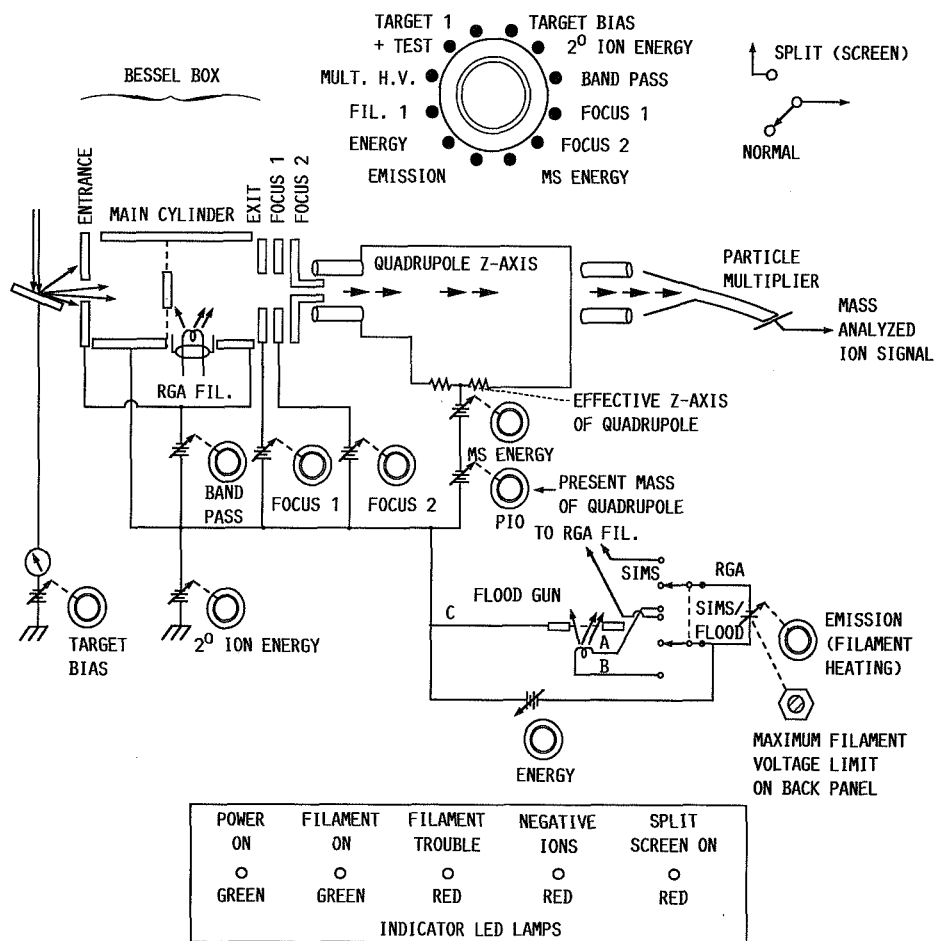


FIGURE 7. - SCHEMATIC OF THE SECONDARY ION OPTICS AND MASS SELECTOR. TAKEN FROM 616-1 MANUAL, WITH PERMISSION FROM EXTREL CORPORATION.

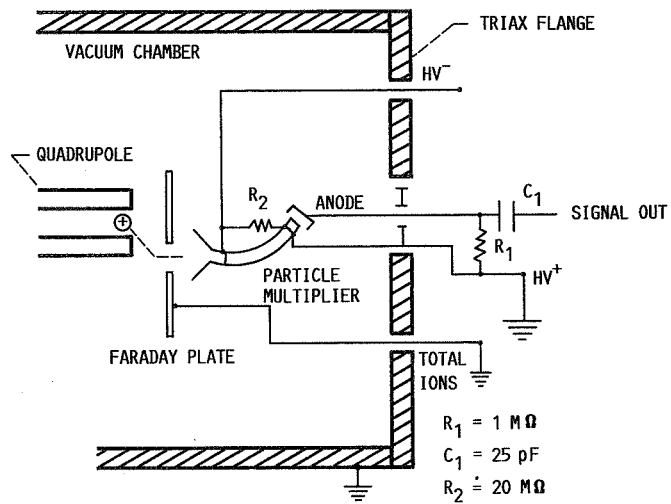


FIGURE 8. - ELECTRON MULTIPLIER CIRCUIT FOR TOTAL-ION COLLECTION.

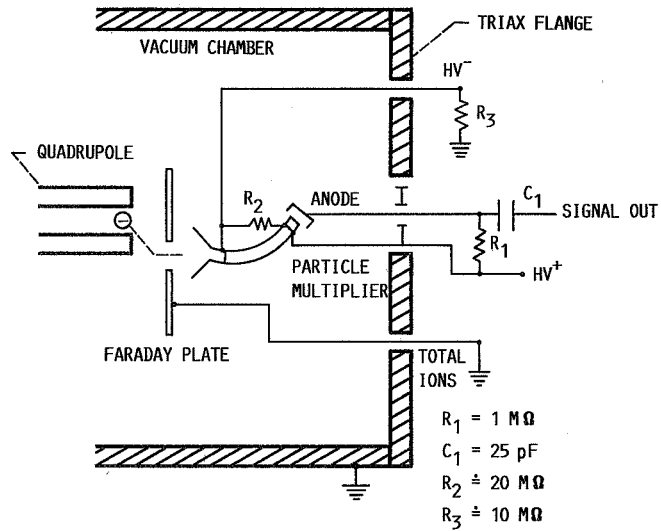


FIGURE 9. - ELECTRON MULTIPLIER CIRCUIT FOR NEGATIVE ION COUNTING.

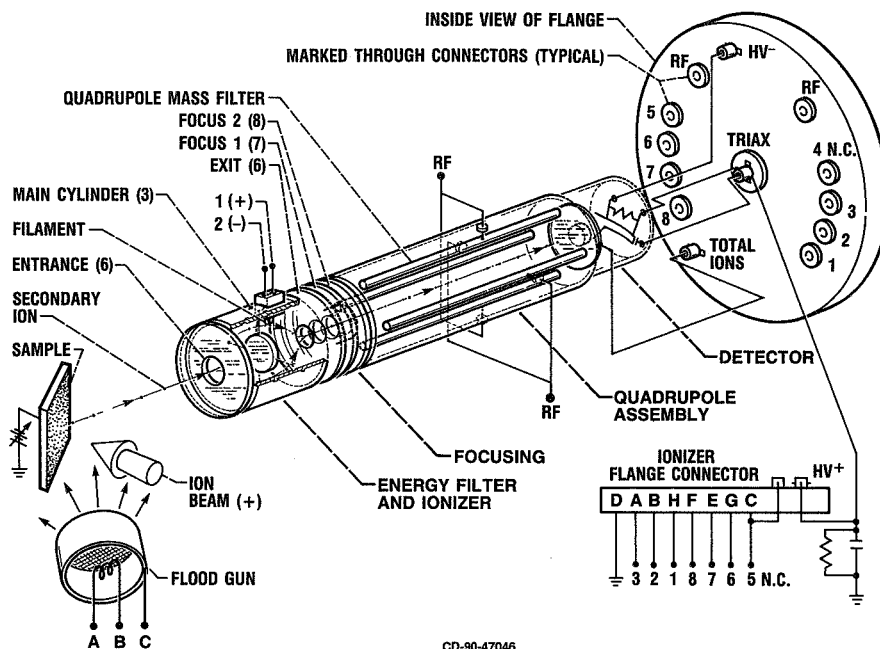


FIGURE 10. - INTERNAL CONNECTIONS TO THE ENERGY FILTER, MASS FILTER AND ELECTRON MULTIPLIER.

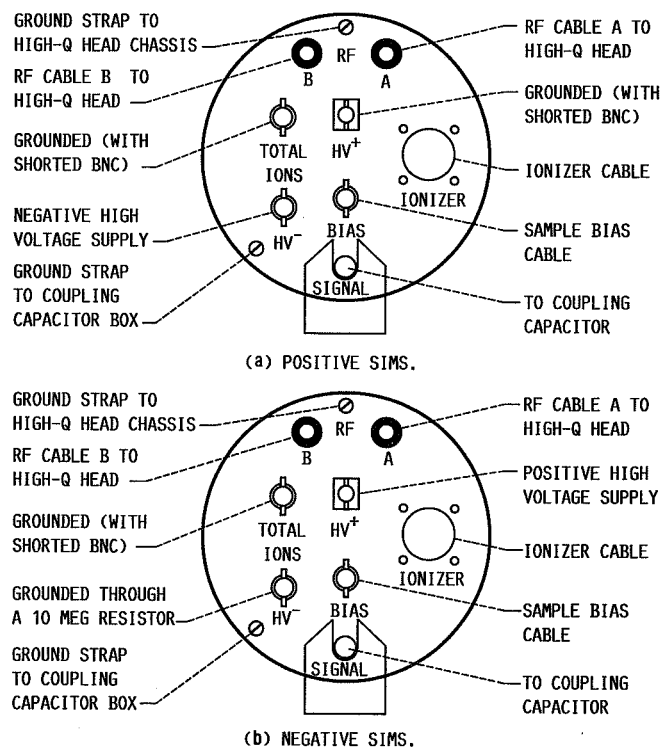


FIGURE 11. - EXTERNAL VIEW OF THE TRIAX FLANGE.

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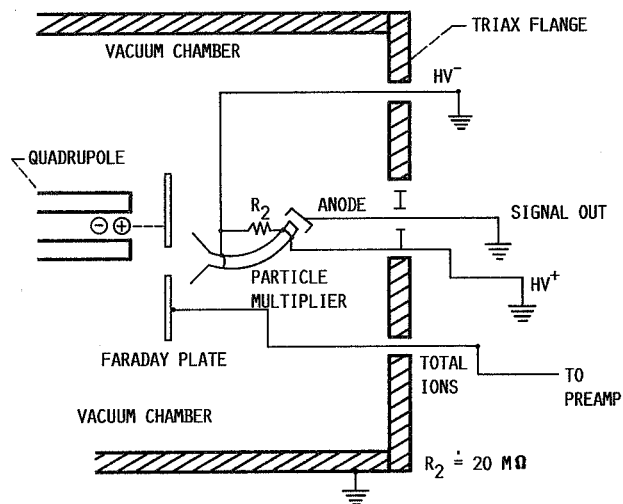


FIGURE 12. - ELECTRON MULTIPLIER CIRCUIT FOR TOTAL-ION COLLECTION.

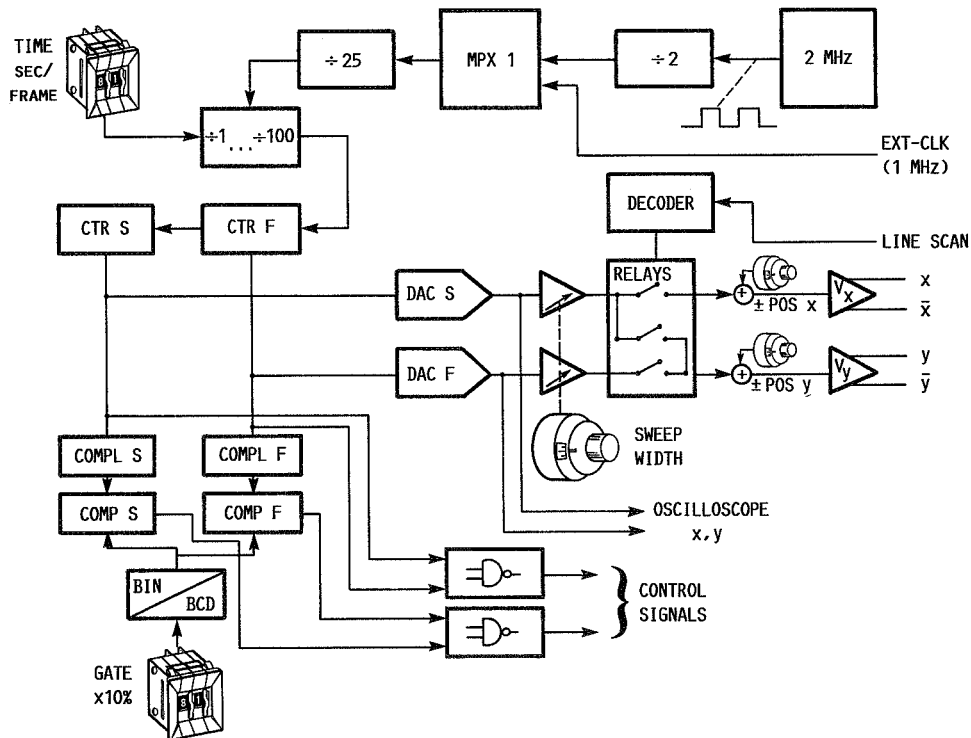


FIGURE 13. - BLOCK DIAGRAM OF THE SCANNING UNIT.

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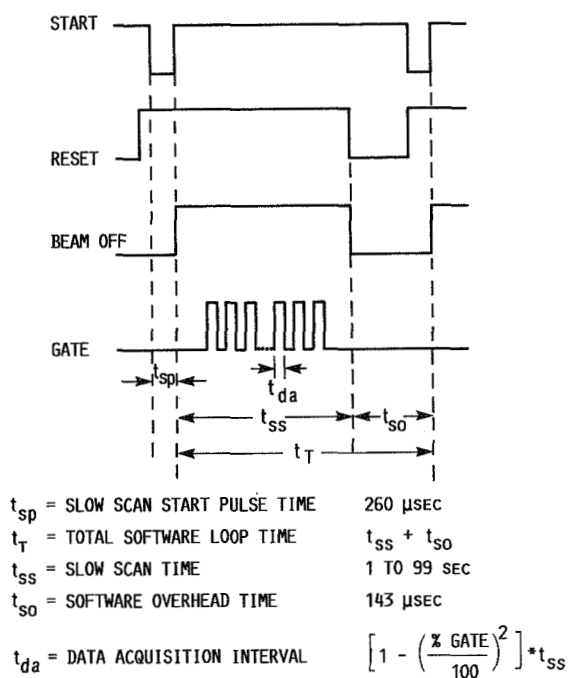


FIGURE 14(a). - TIMING DIAGRAM FOR CONTROL OF SCANNING UNIT IN THE "SINGLE" MODE.

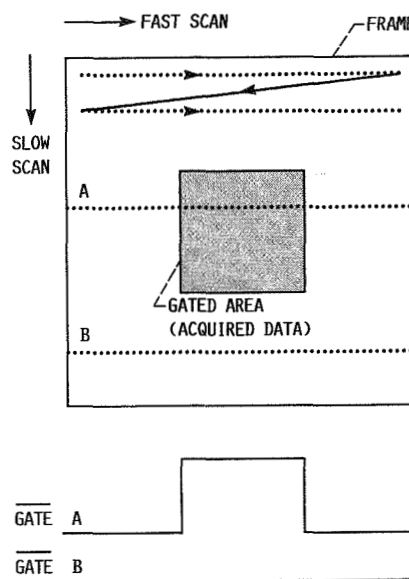


FIGURE 14(b). - DETAIL OF THE BEAM SCANNING PATTERN ON THE SAMPLE AND ITS RELATIONSHIP TO THE GATE SIGNAL.

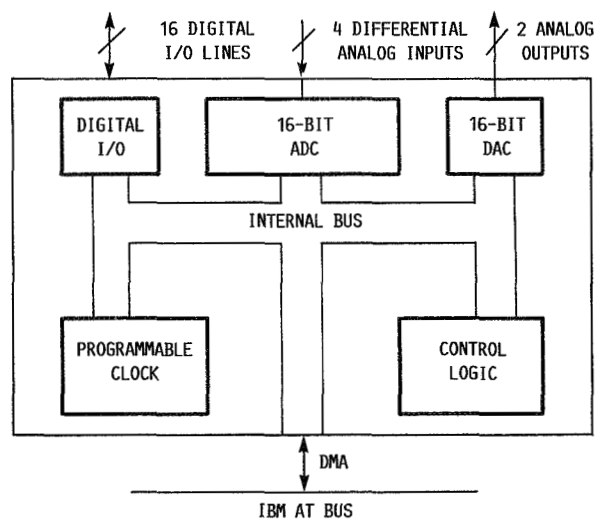


FIGURE 15. - SCHEMATIC DIAGRAM OF THE HIGH-RESOLUTION PLUG-IN BOARD (DT2823) USED TO CONTROL THE ION BEAM AND MASS SPECTROMETER.

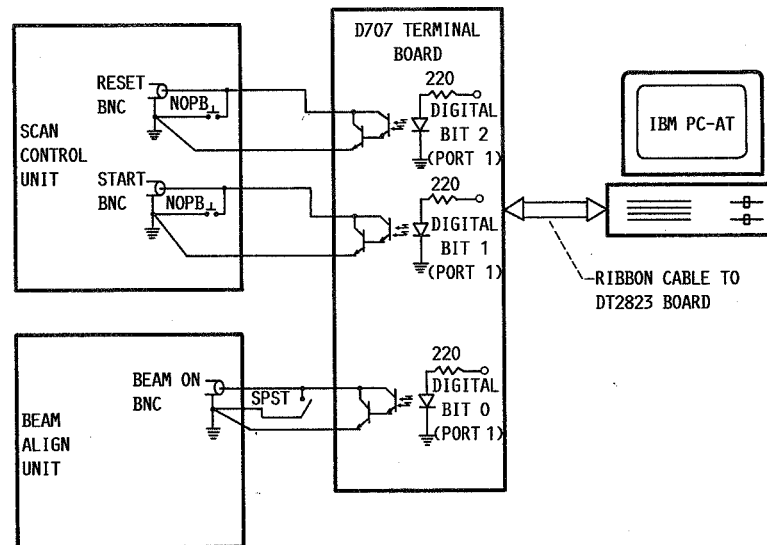


FIGURE 16. - INTERFACE BETWEEN THE PC (VIA THE TERMINAL BOARD OF THE DT2823) AND THE SCANNING AND BEAM ALIGN UNITS.

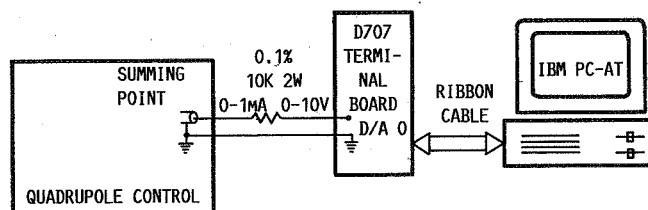


FIGURE 17. - INTERFACE BETWEEN THE PC (VIA THE TERMINAL BOARD OF THE DT2823) AND THE QUADRUPOLE CONTROL UNIT.

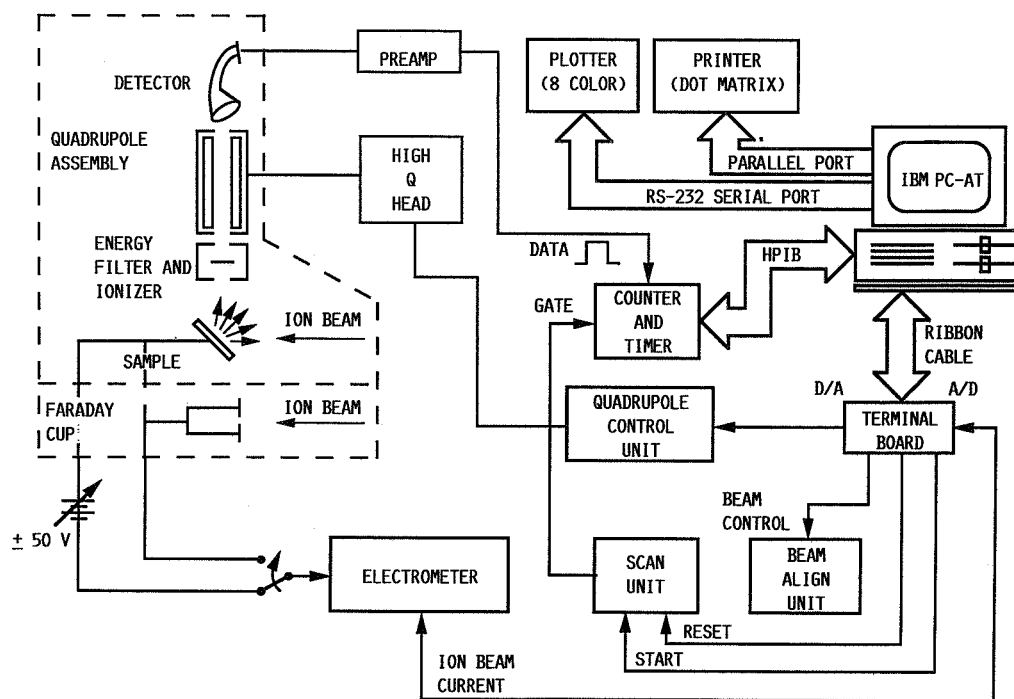


FIGURE 18. - BLOCK DIAGRAM OF THE SIMS SYSTEM AND ITS ASSOCIATED ELECTRONICS.

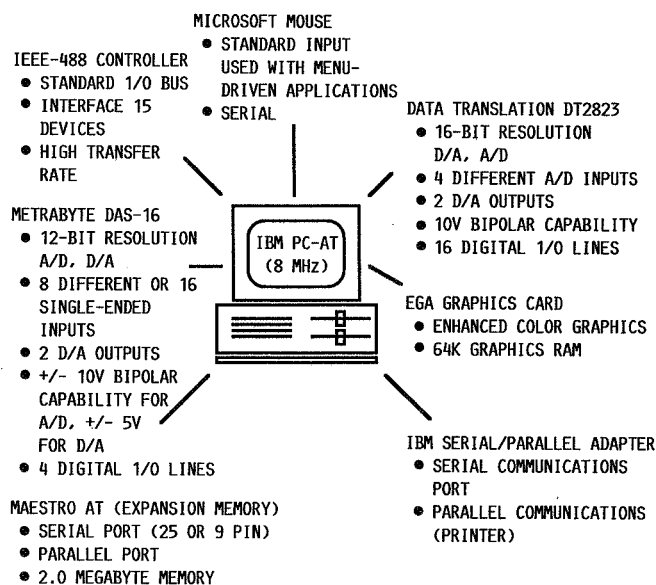


FIGURE 19. - HARDWARE CONFIGURATION OF THE PC.

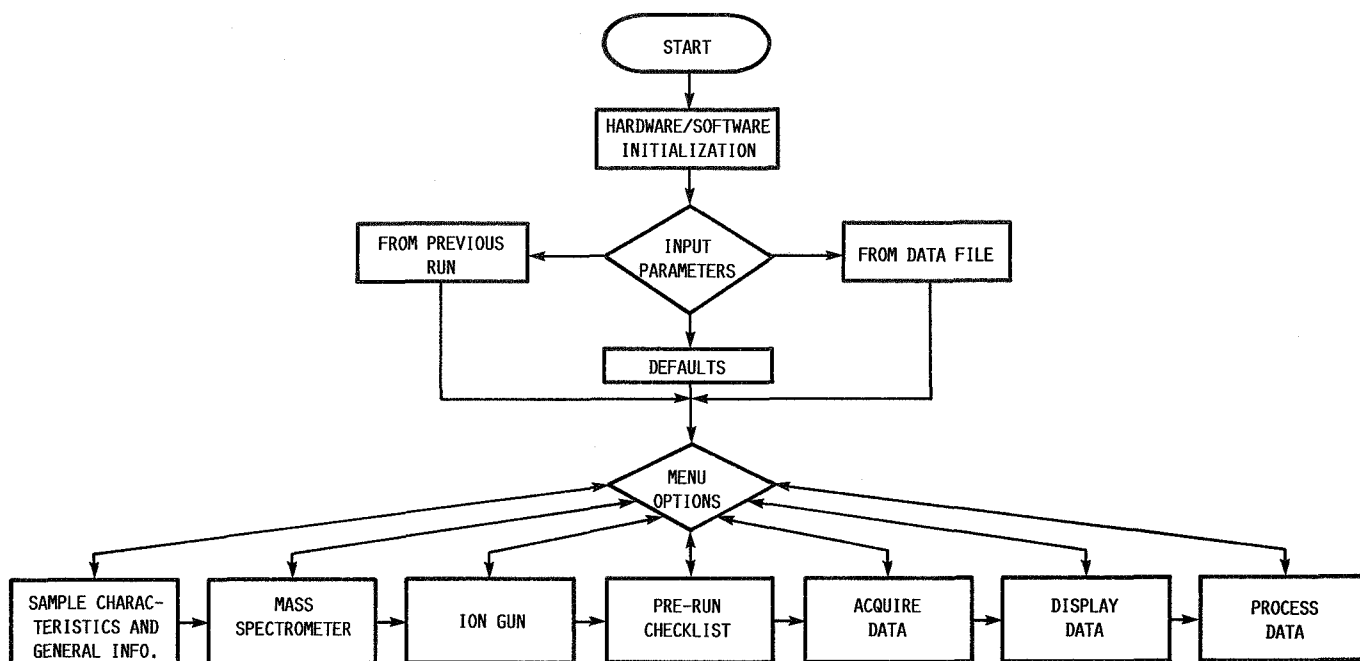
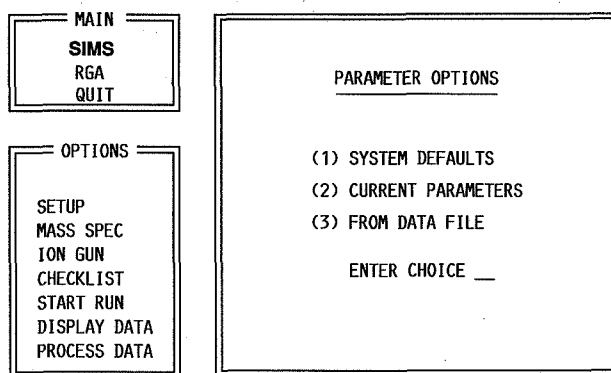
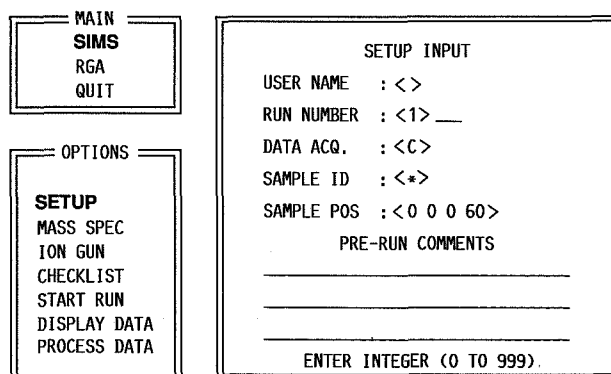


FIGURE 20. - FLOW CHART OF THE MENU-DRIVEN DATA ACQUISITION AND CONTROL PACKAGE.



(a) MAIN SCREEN FOR SIMS ANALYSIS.



(b) INPUT SCREEN FOR SETUP OPTION IN SIMS ANALYSIS.

FIGURE 21.

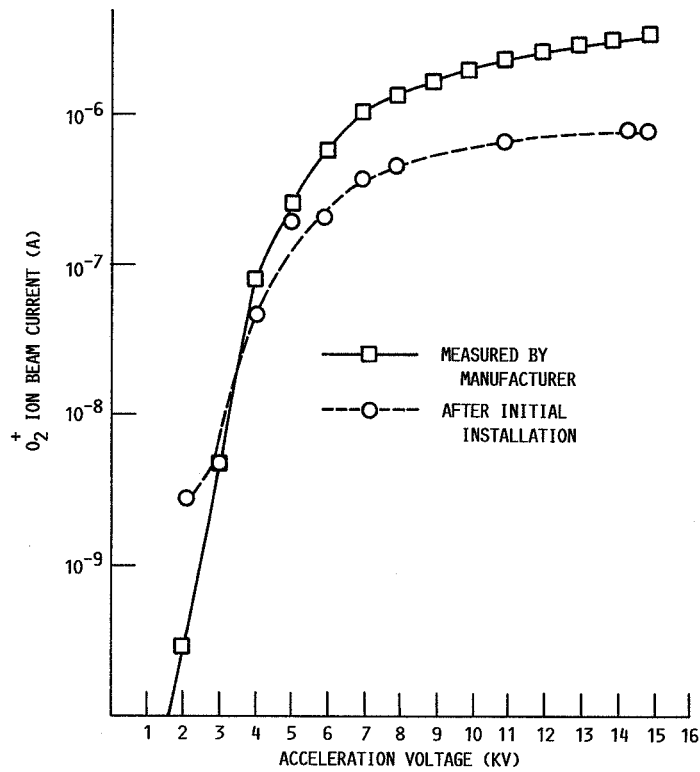


FIGURE 22. - PLOT OF THE MAXIMUM OXYGEN BEAM CURRENT AS A FUNCTION OF ACCELERATING POTENTIAL FOR THE ION GUN.

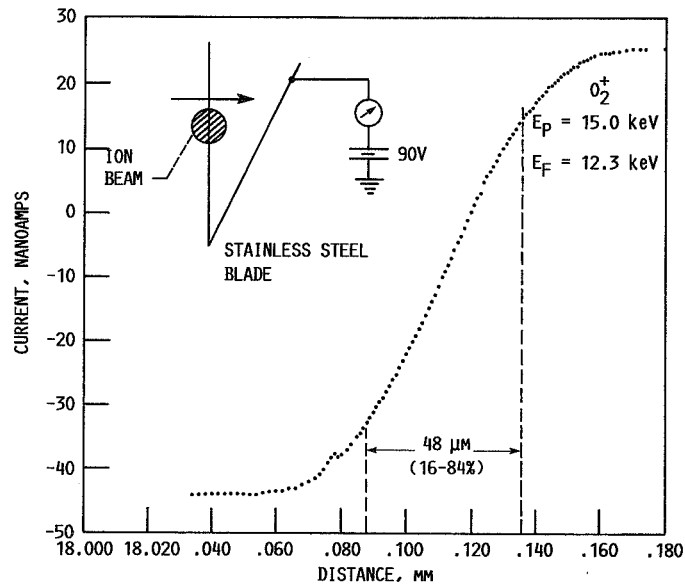


FIGURE 23. - PRIMARY ION (OXYGEN) BEAM PROFILE FOR A BEAM CURRENT OF 75 NANOAMPERES. MEASUREMENT TECHNIQUE IS SHOWN IN THE INSET.

BEAM DIAMETER $\approx 8 \mu\text{m}$ (16-84%)
 BEAM CURRENT = 1 na, ENERGY = 12 keV

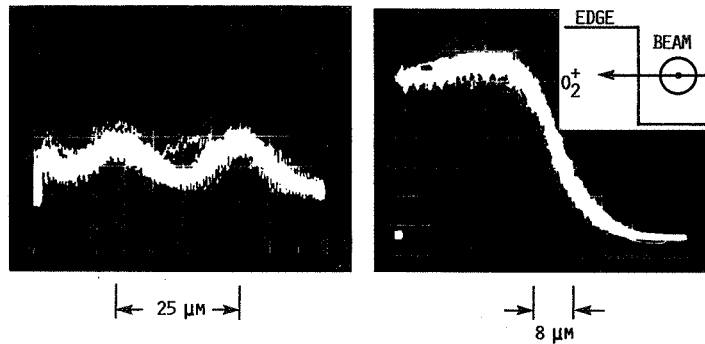


FIGURE 24. - PRIMARY ION (OXYGEN) BEAM PROFILE FOR A BEAM CURRENT OF 1 NANOAMPERE.

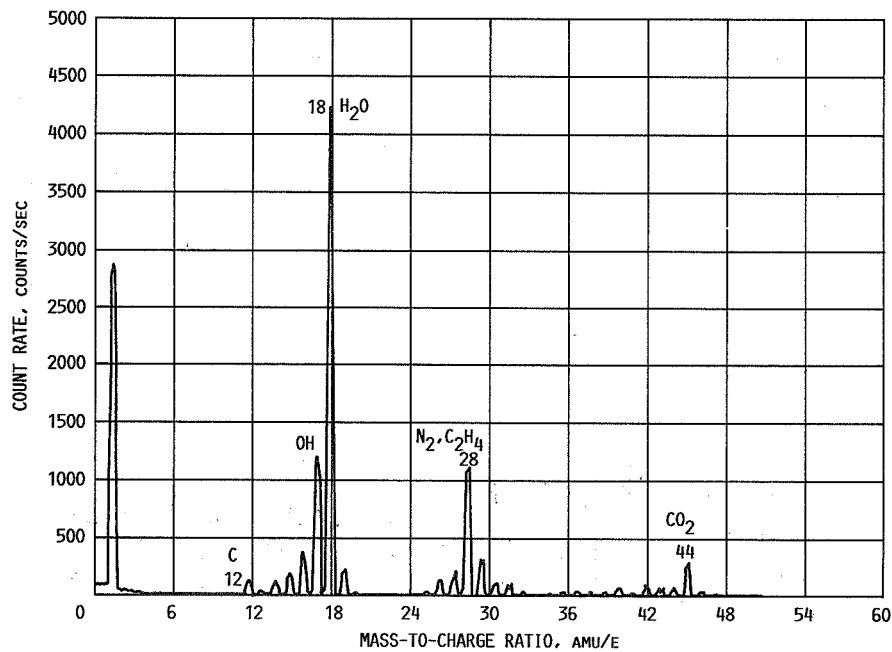


FIGURE 25. - TYPICAL RGA SPECTRUM OF THE UHV CHAMBER ($p = 2 \times 10^{-10}$ MBAR)

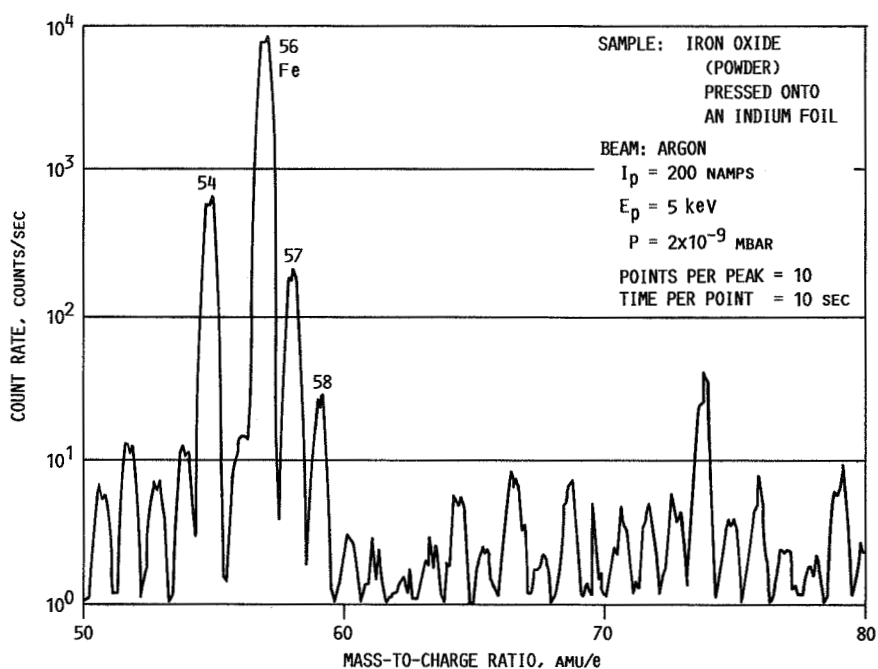


FIGURE 26. - POSITIVE SIMS SPECTRUM OF AN IRON OXIDE (Fe_2O_3) POWDER SAMPLE (PRESSED ONTO AN INDIUM FOIL FOR SUPPORT). 5 keV ARGON PRIMARY BEAM.

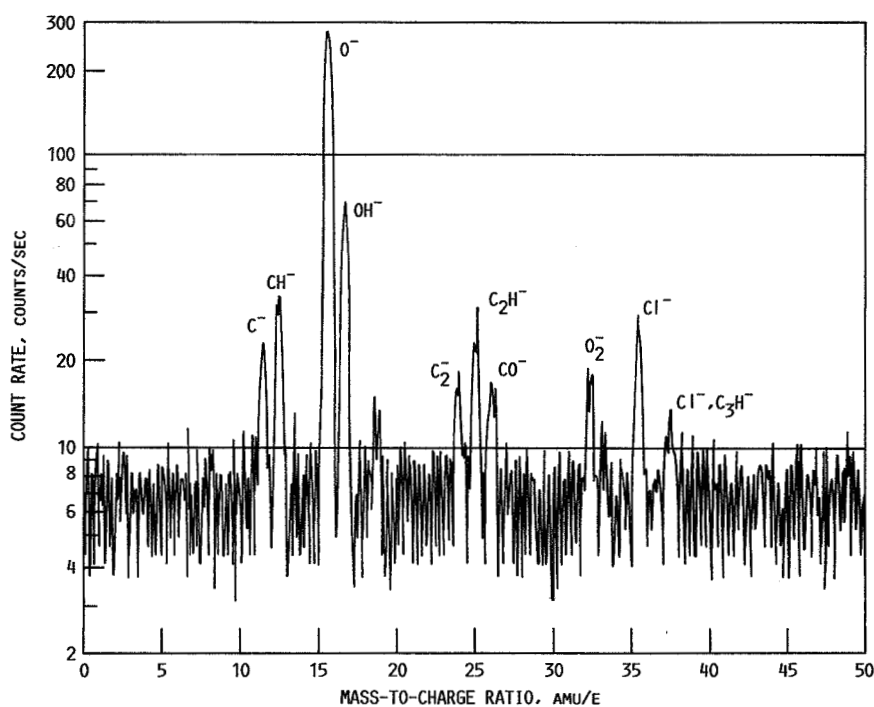


FIGURE 27. - NEGATIVE SIMS SPECTRUM OF A STAINLESS STEEL SAMPLE. 5 keV ARGON PRIMARY BEAM.

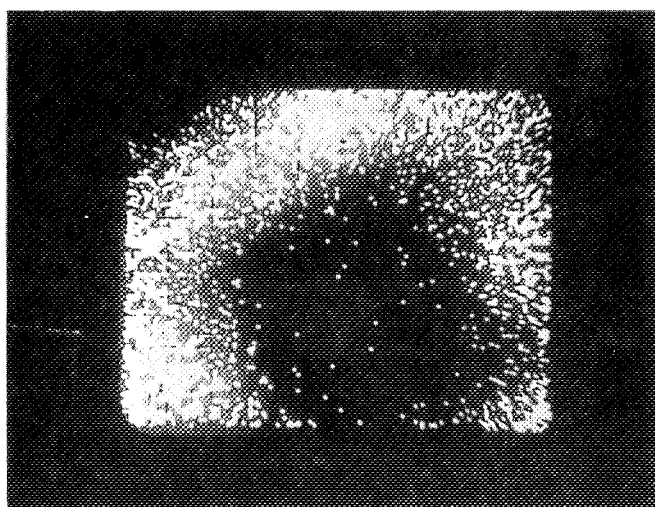
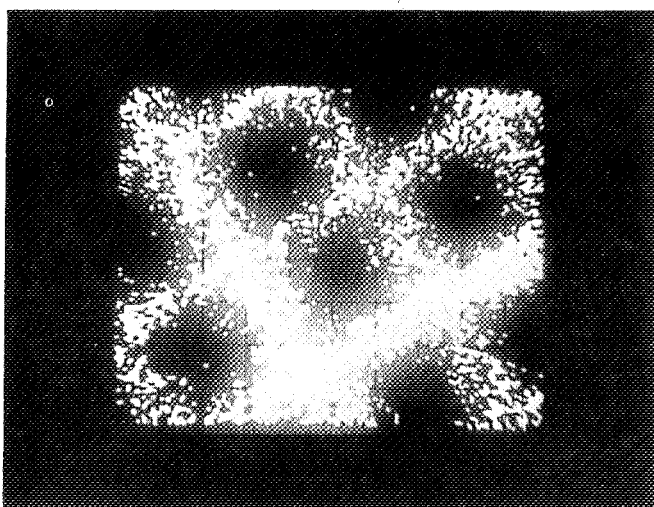
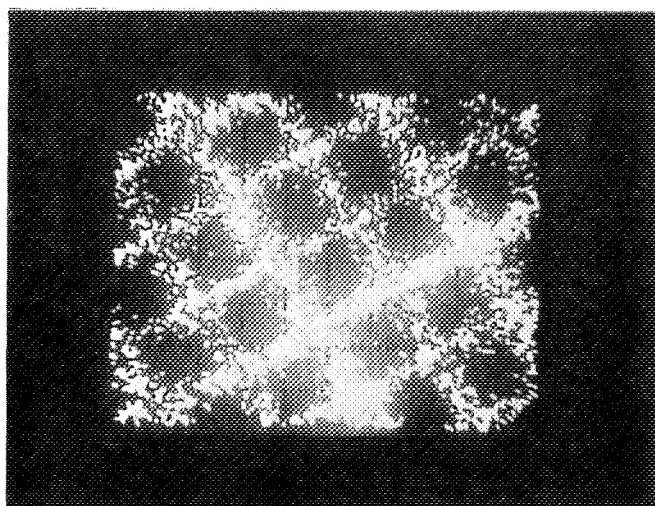
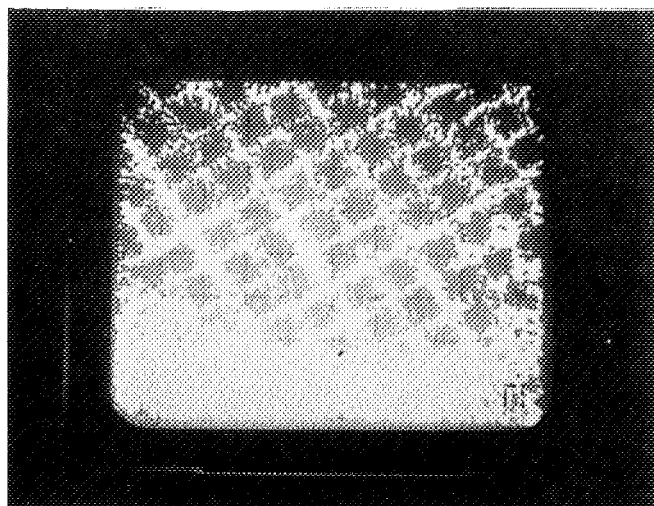


FIGURE 28. - Cu IMAGE OF TEM Cu GRID AS A FUNCTION OF SCAN WIDTH. DISTANCE BETWEEN WIRES IS ABOUT 70 μm .

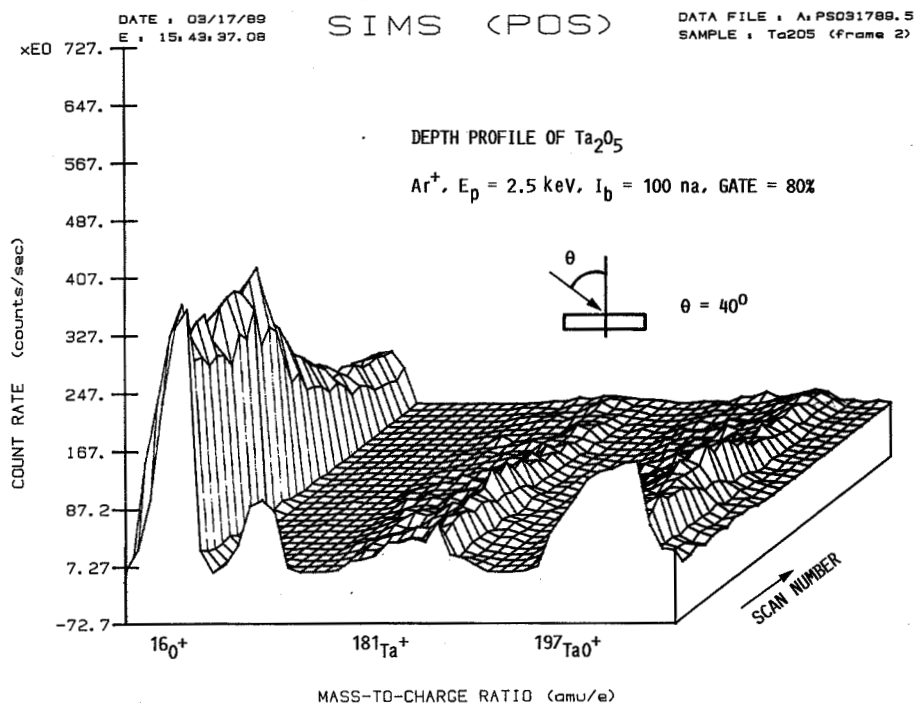


FIGURE 29. - DEPTH PROFILE OF A 1000 Å TANTALUM OXIDE FILM ON A TANTALUM SUBSTRATE.

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Report Documentation Page

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| 15. Supplementary Notes Carlos Vargas-Aburto and Dale R. Liff, Kent State University, Kent, Ohio; Paul R. Aron, NASA Lewis Research Center. | | | | | |
| 16. Abstract <p>We report the design, construction, and initial use of an ion microprobe to carry out secondary ion mass spectrometry (SIMS) of solid samples. The system is composed of a differentially pumped custom-made UHV chamber, a quadrupole mass spectrometer and a telefocus A-DIDA ion gun with the capability of producing beams of Cesium, as well as of inert and reactive gases. The computer control and acquisition of the data have been designed and implemented using a personal computer with plug-in boards, and external circuitry built as required to suit the system needs. The software is being developed by us using a FORTH-like language. Initial tests aimed at characterizing the system, as well as preliminary surface and depth-profiling studies are presently underway.</p> | | | | | |
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